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Indian Standard

PESTICIDES — DETERMINATION OF RESIDUES IN FOODS — FENTHION

भारतीय मानक

कीटनाशी — खाद्य पदार्थों में अवशिष्ट ज्ञात करना — फेंथियॉन

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

September 1989 **Price Group 2**

Pesticides Residue Analysis Sectional Committee, AFDC 56

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards on 23 February 1989 after the draft finalized by the Pesticides Residue Analysis Sectional Committee had been approved by the Agricultural and Food Products Division Council.

Fenthion formulations are extensively used in agriculture and public health for the control of many insect pests. This standard will enable the health authorities and others engaged in the fields to follow uniform test procedure for the estimation of fenthion residue in foods (fruits and vegetables).

In the preparation of this standard, due consideration has been given to the limits of fenthion residues laid under the provisions of *Prevention of Food Adulteration Rules, 1955* and the test method is sensitive to the prescribed level of residues.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*).'

Indian Standard

PESTICIDES — DETERMINATION OF RESIDUES IN FOODS — FENTHION

1 SCOPE

1.1 The standard prescribes gas chromatographic method for the quantitative estimation of fenthion [*o*, *o*-dimethyl-*o* (4-methyl mercapto-3-methyl phenyl)-thiophosphate in foods (fruits and vegetables). Fenthion has to be estimated as its metabolite, fenoxon sulphone.

1.1.1 This method is applicable with a limit of detection in the range of 0·1 μg/g.

1.1.2 Though no set procedure for thin layer chromatography (TLC) is being prescribed, any standardised TLC procedure may be followed if necessary for clean up, identification and confirmation of fenthion residues.

2 REFERENCES

2.1 The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
IS 1070 : 1977	Specification for water for general laboratory use (*second revision*)
IS 11380 : 1985	Methods of sampling for the determination of pesticide residues in agricultural and food commodities

3 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1977) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

4 SAMPLING

The representative samples of foods (fruits and vegetables) for estimating fenthion residues shall be drawn in accordance with IS 11380 : 1985.

5 SAMPLE STORAGE

Samples shall be stored as such when the quantity is small or chances of its degradation do not exist. Otherwise, carry out extraction (*see* **6.4**) and store the extract. Depending upon the nature of the sample, keep the samples or their extracts either in deep freezer at — 15°C or in refrigerator until taken up for analysis. Ensure that the samples do not absorb or lose moisture during storage. Avoid undue long storage periods.

6 GAS CHROMATOGRAPHIC METHOD

6.1 The residues of fenthion are extracted with acetone. The extracted fenthion is converted to fenoxon sulphone and is measured by gas chromatograph equipped with a flame photometric detector. The content of fenoxon sulphone, μg/g (ppm) is determined by comparing the response with that of a known standard fenoxon sulphone of similar concentration.

6.2 Apparatus

6.2.1 *High Speed Blender*, With explosion-proof motor and solvent leak-proof gas jar.

6.2.2 *Rotary Vacuum Evaporator*

6.2.3 *Laboratory Shaker*

6.2.4 *Buchner Funnel Assembly for vacuum filtration*

6.2.5 *Chromatography Columns*, 500 mm length and 18 mm ID.

6.3 Reagents

6.3.1 *Acetone*, glass re-distilled.

6.3.2 *Chloroform*, glass re-distilled.

6.3.3 *Benzene*, glass re-distilled.

6.3.4 *Magnesium Sulphate Solution*, 20 percent (*m*/*v*).

6.3.5 *Potassium Permanganate Solution*, 0·1 M.

6.3.6 *Silica gel*, 60-120 mesh, column chromatography grade. Activated at 110°C for 6 hours.

6.3.7 *Sodium Sulphate*, anhydrous

6.3.8 *Hyflow Supercell filter-aid*, or its equivalent.

6.3.9 *Precipitating Solution*

Dissolve 25 g ammonium chloride and 25 g of phosphoric acid in approximately 900 ml of water and dilute further to 1 litre.

6.3.10 *Fenoxon Sulphone*

Weigh accurately about 200 mg of fenoxon sulphone of known purity into a 100-ml volumetric flask, add acetone and shake well to dissolve the fenoxon sulphone and make up the volume to the mark with acetone. Take 1 ml of this solution into another 100-ml volumetric flask and dilute upto the mark with acetone. The final solution will have the concentration of 0·02 mg of fenoxon sulphone per millilitre of acetone.

6.3.10.1 *Oxidation of standard fenthion of known purity will also produce fenoxon sulphone*

Prepare a solution containing 0·02 mg of fenthion (of known purity) per millilitre of acetone. Take a 2-ml portion of this solution into a 50-ml round-bottom flask. Add 5 ml of 20 percent magnesium sulphate solution and 20 ml of 0·1 m potassium permanganate solution, washing down the sides of the flask during additions and allow to stand for 20 minutes with occassional swirling. Make sure that there is an excess of potassium po manganate throughout the oxidation period. Transfer the oxidised material to a 100-ml separating funnel. Rinse the flask with 50 ml of chloroform and add to the separating funnel. Shake well and allow to stand. Drain the lower organic phase into a round-bottom flask through anhydrous sodium sulphate. Repeat the extraction twice with 50 ml chloroform each time and evaporate the chloroform layer on a rotary vacuum evaporator below 45°C. The fenoxon sulphone is transferred into a 2-ml volumetric flask with acetone and diluting the solution up to the mark.

6.4 Preparation of Sample, Clean-up and Extraction of Fenthion Residues

6.4.1 Transfer 100 g of the sample to a waring blender and crush at high speed for 2-3 minutes. To this add 150 ml acetone and 100 g of powdered anhydrous sodium sulphate. Blend for 2 minutes and allow to settle for one minute. Decant the solvent into a Buchner funnel attached to a 500-ml filter flask and containing Whatman No. 1 filter paper with a thin coat of hyflo supercel or equivalent filter aid. Apply vacuum cautiously until all the solvent has filtered into the filtration flask. Re-extract the sample with 2 × 100 ml portions of acetone and filter the extracts. Rinse with 50 ml acetone. Transfer the filtrates to a round-bottom flask and distill off the solvent just to dryness on a rotary vacuum evaporator below 45°C.

6.4.2 Dissolve the residue in 45 ml acetone and add to it 50 ml of precipitating solution. Allow the mixture to stand for 30 minutes with occassional swirling. Filter using vacuum through 3 mm layer of hyflo supercel on Whatman No. 1 filter paper in a Buchner funnel into a receiving flask. Rinse the flask and wash the residue 2 × 30 ml with a mixture of acetone : precipitating solution (45 : 50), allowing each washing to remain in contact with precipitate for about 20 seconds before applying vacuum. Transfer the filtrate to a 250-ml separating funnel and extract it with 125, 100 and 100 ml of Chloroform. Pass the combined organic solvent phase through anhydrous sodium sulfate and collect it in 1000-ml round-bottom flask. Distill off the solvent on a rotary vacuum evaporator.

6.5 Oxidation

6.5.1 Dissolve the sample residue obtained after completion of step **6.4.2** in 2 ml of acetone. Add 5 ml of 20 percent magnesium sulphate solution and 20 ml of 0·1 M potassium permenganate solution, washing down the sides of the flask during additions and allow to stand for 20 minutes with occassional swirling. Make sure that there is an excess of potassium permanaganate throughout the oxidation period. Transfer the oxidised material to a 100-ml separating funnel. Rinse the flask with 50 ml of chloroform and add to the separating funnel. Shake well and allow to stand. Drain the lower organic phase into a round-bottom flask through anhydrous sodium sulphate. Repeat the extraction with 2 × 50 ml and evaporate the collected chloroform layer on a rotary vacuum evaporator below 45°C.

6.5.2 *Clean up for oxidised extract by column chromatography*

Prepare a chromatography column (*see* **6.2.5**) using 10 g of activated neutral silica gel and 95 : 5 benzene acetone mixture. Rinse the flask containing the residue with 3 × 3 ml portions of solvent mixture and transfer the extract to the column. Elute the column with 75 ml of solvent mixture at the rate of 8 to 10 ml per minnte. After removal of the solvent on a rotary evaporator below 45°C, the residue is redissolved in acetone and quantitatively transferred to a 5-ml volumetric flask. Dilute upto the mark and shake well to mix.

6.6 Gas Chromatograph

A gas Chromatograph equipped with a flame photometric detector is operated under the following suggested parameters. The parameters may be varied according to the available facilities, provided standardisation is done:

Column	glass, 90 cm long, 6·25 mm diameter
Packing	5%OV — 101 on Chromosorb G HP 80 — 100 mesh
Carrier gas	Nitrogen
Carrier gas flow rate	60 ml/min
Combustion gas and flow rate	Hydrogen, 200 ml/min; Oxygen, 30 ml/min; Air 100 ml/min
Column temperature	230°C

Injection Port Temperature	250°C
Detector temperature	250°C
Detector	flame photometric detector operating in phosphorus mode

6.7 Procedure

Inject a suitable aliquot (3 μl) into the column using a 10-microlitre syringe. Indentify the peak by its retention time and measure the peak area.

6.8 Calculations

Fenthion, μg/g (ppm)

$$= \frac{A_1 \times V_2 \times V_3 \times C}{A_2 \times V_1 \times M} \times f \times \frac{278}{310}$$

where

A_1 = Peak area of sample;

V_2 = Volume in μl of standard injected;

V_3 = Total volume in ml of sample solution;

C = Concentration of standard solution, in μg/g;

A_2 = Peak area of standard;

V_1 = Volume in μl of sample injected;

M = Mass in g of sample taken for extraction; and

F = Recovery factor

$$= \frac{100}{\text{Percent mean recovery}}$$

NOTES

1 $\frac{278}{310}$ is the conversion factor of fenoxon sulphone to fenthion.

2 Percent mean recovery is determined by taking untreated control sample to which a known amount of fenoxon sulphone is added and analysed as described above.

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